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MICROSTRUCTURAL CHARACTERIZATION OF A COBALT-FREE MARAGING STEEL, VASCO MAX T-250

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MATERIALS CHARACTERIZATION DIVISION

February 1987

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ABSTRACT

A cobalt-free maraging steel, Vasco Max T-250, is being used as a replacement for the conventional, 18% Ni 300 grade maraging steel used by the Army. The thrust of this program was to characterize the microstructure of annealed and aged specimens using optical and electron microscopy and X-ray diffraction.

The structure of the annealed sample is a heavily dislocated lath martensite. The aged sample are a lath martensite that contains very fine Ni_3Ti second phase particles. The precipitates are coherent with the matrix and form in a Widmanstatten pattern. The Ni_3Ti precipitates are a very effective strengthening phase for the Vasco Max T-250 and the aging temperature that appears to optimize the strengthening precipitates is 950°F. Keywords,

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INTRODUCTION

Maraging steels are ultrahigh strength steels which, in contrast to most other types of steel, have a very low concentration of carbon. They employ substitutional elements such as nickel, molybdenum, and titanium to achieve an age-hardened martensitic structure rather than the usual quench-and-tempered structure. These steels possess one of the highest combinations of strength and fracture toughness on any commercially available alloy.

Maraging steels contain a high concentration of nickel (usually 20% to 25%) which ensures a complete transformation to martensite even with a very slow cool from the austenization temperature. There is an increase in the thermal hysteresis between the formation of martensite on cooling and austensite reversion on heating which allows the aging of the martensite matrix at elevated temperatures (around 850°F to 950°F).[1] The precipitation reactions that occur upon aging the martensitic matrix are mainly responsible for the ultrahigh strengths, hence the term "maraging."

PURPOSE

A conventional 18% Ni 300 grade maraging steel was used by the Army for missile motor cases. The conventional grades of maraging steels contain between 8% and 15% cobalt. Cobalt is a strategic and critical material because it has essential defense related uses and the U S. must import its entire supply. Therefore, a cobalt free maraging steel, Vasco Max T-250, is being studied as a replacement for the cobalt containing 18% Ni 300 grade maraging steel.

The investigation reported in this paper was undertaken to characterize the microstructure of the Vasco Max T-250, and determine the structural changes that occur during aging.

BACKGROUND

Alloying iron with a high concentration of nickel ensures a total martensitic structure, even with very slow cooling from the solution annealing temperature by delaying equilibrium phase nucleation. In addition, the presence of nickel also reduces the solubility of many other elements (Ti, Mo, Al, etc.) in iron. Lath martensite forms upon cooling due to the low carbon concentration.[2] The martensite laths act as barriers to slip resulting in increased strength which can be approximated by the familiar Hall-Petch[3,4] relationship:

$$\sigma_y = \sigma_o + Kd^{-\frac{1}{2}}$$

where

d = average lath size
σy = yield strength
σ_o and K = material constants.

The structure of maraging steels after annealing consists of a series of elongated laths or platelets that contain a high density of dislocations.[5,6] The dislocations within the massive martensitic structure tend to be predominantly screw in nature.[7]

It is generally known that the crystal structure of lath martensite is bcc.[2] Bcc crystals usually slip on the {110} dodecahedral planes and in the <111> cube diagonal directions. Slip may also occur on the {112} and {123} planes. Even though there are a total of 48 possible slip systems, bcc crystals have a relatively high Peierls-Nabarro stress and therefore have few mobile dislocations.[8] However, the presence of nickel slightly offsets this effect since nickel reduces the resistance of the crystal lattice to disloaction motion.[9]

The introduction of dislocations into the steel by means of the metastable phase transformation increases the flow stress. The dependence of the flow stress on the dislocation density can be determined by:

$$\tau = \alpha\ G b \sqrt{\rho} + \tau_0 \qquad \text{Ref. 10)}$$

where

τ = flow stress
τ_0 = "friction stress"
α = 0.5, a dimensionless constant
G = the shear modulus
b = the Burgers vector
ρ = dislocation density.

The high dislocation densities found in maraging steels, 10^{12} cm^{-2}, produce a dramatic strengthening effect by increasing the flow stress.

The hardening induced during aging of conventional maraging steels results from the following two mechanisms:

1. The precipitation of various intermetallic compounds that are coherent with the matrix, and

2. Solid solution strengthening due to the long range order interaction of cobalt.

Many types of precipitates have been reported in the literature. A considerable effort has been directed at characterizing the phases precipitated during aging and the morphology and distribution of these precipitates. Techniques employed in these investigations were mainly electron microscopy and X-ray diffraction analysis.

A literature search of the precipitate characterization studies reveals discrepancies in the phases, structure, and morphology. This can be partially attributed to the uncertainties inherent in the techniques employed and the small particle size. The shape of the precipitates has been reported to be spherical,[11-15] disk shaped,[16] ribbon shaped,[14] or needlelike.[13,14,17]

The most frequently reported particle is Ni_3Mo. This precipitate is rod shaped, having a width of 25 Å and a length of 500 Å in the peak hardness condition. The longer axis is paralled to the <111> matrix directions. The orientation relationship between Ni_3Mo and the martensite matrix has been shown to have been:

$$(010)\ Ni_3Mo \parallel (011)_\alpha \qquad [100]\ Ni_3Mo \parallel [1\bar{1}1]_\alpha .$$

In other words, the closest packed plane and direction in the matrix are parallel to those in the precipitates.

Ni_3Ti is the intermetallic compound most often reported for the titanium-rich precipitates. There is some evidence that suggests that preprecipitate zones may form during the early stages of aging. Diffraction streaks have been observed in titanium-rich maraging steel after aging for only two minutes at 950°F, suggesting that there is a G.P. zone stage preceeding precipitation.[13] Diffraction patterns from thin foils were analyzed and the following relationship between the matrix and precipitate was developed:

$$(011)_\alpha \parallel (0001)\ Ni_3Ti \qquad [11\bar{1}]_\alpha \parallel [11\bar{2}0]\ Ni_3Ti.$$

These precipitates form parallel to the <111> matrix directions.[18] The following mechanism had been proposed: martensite metastable bcc ordered Ni_3Ti zones (DO_3 structure) Widmanstatten [precipitation of stable Ni_3Ti phase (DO_{24} structure)].

Irrespective of the precipitate origin, these particles impede dislocation motion. The precipitates are shearable by dislocations, which enhances their strengthening mechanism. Hardening is due to coherency strains, ordered structure, and interfacial energy.[12,19,20,21]

Precipitates formed during aging have been reported to have nucleate both at martensite latn boundaries and at dislocations. The heavy dislocation density provides a uniform distribution of precipitate nucleation sites and, as a result, the precipitates are uniformly distributed. Large precipitates or precipitate-free zones at grain boundaries are not normally found. The precipitates nucleate and grow with a preferred orientation with respect to the matrix due to the preferred orientation of the dislocations within the martensite matrix.[7] Precipitation usually occurs along the lengths of the dislocations, therefore precipitates lie in the <111> directions.[13]

ROLE OF COBALT

In conventional maraging steels, cobalt, in combination with molybdenum, makes an important contribution to the strengthening of maraging steels. It is generally believed that cobalt lowers the solubility of molybdenum in the martensite matrix, therefore, favoring the precipitation of molybdenum containing intermetallic compounds.[17,22,23] It has also been suggested that cobalt may affect the dislocation substructure in the matrix, ultimately providing more uniformly distributed nucleation sites for precipitation.[22,25] The removal of cobalt from maraging steels would increase the solubility of molybdenum in the martensite matrix and decrease the probability of molybdenum precipitating to form an intermetallic compound. Therefore, the cobalt-free maraging steels are most likely hardened by precipitates that do not contain molybdenum.

EXPERIMENTAL

A supply of cobalt-free maraging steel, Vasco Max T-250, was received from Teledyne Vasco in the form of rolled 3-inch-diameter bar in the annealed condition. The composition of Vasco Max T-250 is 76.74% Fe, 18.50% Ni, 3.0% Mo, 1.40% Ti, 0.10% Al, 0.01% Zr, 0.003% B, and not more than 0.10% Si and Mn, 0.03% C, and 0.01% S and P. This composition differs from that of a conventional maraging steel because it does not contain cobalt, but contains an increased amount of titanium, and a decreased amount of molybdenum.

At MTL the Vasco Max T-250 solution was annealed at 1500°F and air cooled. The annealed Vasco Max T-250 was aged at 850°F, 900°F, and 950° for 3, 4, and 8 hours. The solution annealed and aged specimens were analyzed using X-ray diffraction, optical microscopy, and transmission electron microscopy. Foils for electron microscopy were prepared by chemical thinning using a Fischione Twin Jet Polisher, with a 20% perchloric 80% methanol polishing solution. The major effort of this program was to identify the phases precipitated during the aging of the martensite and determine the shape, size, and distribution of the precipitates.

RESULTS AND DISCUSSION

X-Ray Diffraction

X-ray diffraction analysis was used to determine the phases present in the annealed and aged samples. Diffraction patterns of the annealed and aged samples were composed of peaks from the bcc martensite phase. The X-ray diffraction analysis did not provide any information on the identity of the second phases present after the aging treatments. The particles could not be identified using X-ray diffraction either because the crystallite size was too small or, because they were not present in sufficient quantity.

Optical Microscopy

The annealed and aged specimens of the Vasco Max T-250 were examined using optical microscopy. The structure of the annealed and aged samples appeared the same, namely a lath martensite. An optical micrograph of the annealed sample is shown in Figure 1. Using optical microscopy, all of the samples appear the same, and it is difficult to distinguish any differences between the annealed and aged structures.

Electron Microscopy

The annealed and aged specimens of the Vasco Max T-250 were examined using a JEOL 200 CX scanning transmission electron microscope. At low magnifications, the structure of the specimens is a typical lath martensite. At higher magnifications, above 20 kx, differences in the structures, such as dislocation density and precipitate distribution, were observed.

A centered, dark field technique was used to image the dislocations and precipitates. The dark field technique consists of tilting the incident illumination so that the diffracted electrons travel along the optic axis. An objective aperture is inserted into the electron column to allow only the diffracted beam to form the image.

Annealed Sample

The annealed sample was air cooled from 1500°F, and the resulting microstructure was a bcc martensite which transformed by diffusionless shear. The microstructure of the annealed sample is shown in Figure 2; it is a typical lath martensite. The elonged laths contain a high density of dislocations, as shown in Figure 3.

Precipitation of second phase particles occurred during the aging of the Vasco Max T-250. The aging temperature greatly influenced the precipitate size and distribution.

850°F AGING TREATMENT

In general, the 850°F aging treatment was below the optimum aging temperature for the T-250 maraging steel because the precipitate density was very low even after the 8-hour age. The structure of the specimen aged at 850°F for 3 hours is shown in Figure 4. It is a heavily dislocated lath structure that is typical of all samples aged at 850°F. A high magnification micrograph of the sample aged at 850°F for 3 hours is shown in Figure 5. It shows the high dislocation density within the laths. Precipitates are not visible in the bright field in the microstructure of the samples aged at this temperature. Using the centered dark field technique and imaging weakly diffracted beams, precipitates were observed in these samples. Dark field micrographs of samples aged at 850°F for 3, 4, and 8 hours are shown in Figure 6. The sample aged for 3 hours contains a very low density of fine precipitates. The precipitates appear needlelike and have a slight directionality. The average size of the precipitates after a 3-hour age is 100 Å long and 30 Å wide. The sample aged for 4 hours also has a low density of fine precipitates. The average size of the precipitates is 100 Å long and 30 Å wide. The sample aged for 8 hours at 850°F has a slightly greater density and larger size precipitates than the samples aged for 3 and 4 hours. The precipitates present after an 8-hour age are needlelike and have an average size of 150 Å long and 30 Å wide.

900°F AGING TREATMENT

Aging the T-250 maraging steel at 900°F enabled more precipitation to occur. The lath martensite structure of the sample aged for 4 hours is shown in Figure 7. Precipitates are visible in the bright field micrographs. A high magnification micrograph of this sample is shown in Figure 8. Dark field micrographs of the samples aged at 900°F for 3, 4, and 8 hours are shown in Figure 9. After a 3-hour age, the average precipitate size is 200 Å long and 50 Å wide. They are very directional and seem to form a Widmanstatten pattern. After the 4-hour age, the average precipitate size is 250 Å long and 50 Å wide. The average precipitate size after an 8-hour age is 250 Å long and 50 Å wide. The needlelike precipitates resulting from the 900°F are larger and present in a greater density than after an 850° age. The particles precipitated during the 900°F age are uniformly distributed and form a Widmanstatten pattern.

950°F AGING TREATMENT

The aging treatments at 950°F precipitated a heavy density of needlelike or rod-shaped particles. The precipitates are visible in bright field and dark field imaging modes. The lath structure typical of the 950°F aging treatment is shown in Figure 10. A higher magnification micrograph of the sample aged for 4 hours is shown in Figure 11. Dark field micrographs of the samples aged for 3, 4, and 8 hours are shown in Figure 12. The average precipitate size after a 3-hour age is 350 Å long and 50 Å wide. There is a high density of precipitates in the sample and they are spaced widely apart. After a 4- and 8-hour age, the average precipitate size is 450 Å long and 70 Å wide. The precipitates are widely spaced and form a Widmanstatten pattern.

PRECIPITATE MORPHOLOGY

Electron diffraction performed on the aged thin foils identified the precipitates as Ni_3Ti, a hexagonal intermetallic compound commonly found to precipitate from high titanium-containing steels.[13] The Ni_3Ti particles are rodlike or needle-like and form with their axes parallel to the <111> of the martensite matrix. Figure 13 shows a bright field/dark field pair of the sample aged at 900°F for 3 hours. The precipitate cross sections are visible and the micrograph has a <111> martensite direction parallel to the electron beam.

Many diffraction patterns of the aged samples were analyzed, and similar diffraction patterns were obtained for most of the aged samples. A typical electron diffraction pattern from samples aged at 900°F and 950°F is shown in Figure 14. The orientation of the matrix is $(011)_\alpha$ and this demonstrates the relation between the matrix and precipitate: $(011)_\alpha \parallel (0001)_\eta$ and $[11\bar{1}]_\alpha \parallel [11\bar{2}0]_\eta$. A schematic diagram showing the orientation relationship is shown in Figure 15.

The precipitates were identified using electron diffraction of thin foils. Due to the uncertainties inherent in the electron diffraction technique, additional testing should be done to confirm the identity of the precipitates. The precipitates should be extracted from the matrix for further analysis.

The removal of cobalt from the composition of the maraging steel, Vasco Max T-250, increases the solubility of molybdenum in the martensite matrix, thereby decreasing the probability of precipitating molybdenum-containing second-phase particles. The increased titanium content and the decreased molybdenum content of Vasco Max T-250 and the decreased probability of precipitating molybdenum intermetallic compounds favors the precipitation of titanium-containing second-phase particles. Titanium plays a dual role of hardener and refining agent to tie up the residual carbon. The titanium-rich compositions η Ni_3Ti is the intermetallic compound most often found[13] and it forms in a Widmanstatten pattern of a stable DO_{24} structure. It has been suggested that preprecipitate zones may form during the initial stages of age hardening. Metastable bcc ordered Ni_3Ti zones may appear before the η Ni_3Ti becomes stable.[13] This could account for the difficulty of observing and identifying the precipitates in the samples aged at 850°F for 3 and 4 hours.

A summary of the precipitate size and distribution is presented in Table 1. It appears that the largest size and distribution of precipitates is present after the 950°F aging treatments. The dislocation density decreases with an increase in the

Table 1. COBALT-FREE MARAGING STEEL, VASCO MAX T-250

Heat Treatment	Precipitate Size Length (Å)	Width (Å)
Annealed	No Precipitates	
850°F, 3 hours	50-150	30
850°F, 4 hours	50-150	30
850°F, 8 hours	100-200	30
900°F, 3 hours	100-300	50
900°F, 4 hours	100-400	50
900°F, 8 hours	100-400	50
950°F, 3 hours	200-500	50
950°F, 4 hours	200-700	60
950°F, 8 hours	200-700	60

aging temperature and time. The precipitates become more widely spaced and a better defined Widmanstatten pattern develops as the aging temperature increases. As was expected from Fick's first and second laws of diffusion, the aging temperature has a greater influence on the structure of the Vasco Max T-250 than the aging time. The effect of aging time would become more pronounced if the aging times were orders of magnitude longer.

Cobalt-containing maraging steels are usually aged at 900°F for between 3 and 6 hours to achieve peak strength. Aging the conventional maraging steels at temperatures greater than 900°F produces a decrease in the strength because the precipitates become incoherent with the matrix, and reversion to austenite occurs. The Vasco Max T-250 aged at 950°F contained the largest precipitates and the highest density of precipitates. At 950°F, the precipitates are still coherent with the matrix and form a Widmanstatten pattern which inhibits the dislocation motion and increases the strength. Therefore, the 950°F aging treatment is considered the optimum treatment because of precipitate morphology. The cobalt-containing and cobalt-free maraging steels are strengthened by different phases, therefore, the optimum aging temperatures are different. The difference in diffusion rates of the strengthening precipitates determines the optimum aging temperature. Conventional maraging steels are most often strengthened with Ni_3Ti precipitates and the Vasco Max T-250 cobalt-free maraging steel is strengthened with Ni_3Ti precipitates.

CONCLUSION

The microstructure of the Vasco Max T-250, a cobalt-free maraging steel, was characterized in the annealed and aged condition. In the annealed condition, the structure consisted of a heavily dislocated lath martensite. After aging the Vasco Max T-250, very fine second-phase particles precipitated from the lath martensite structure. These second-phase particles were identified through electron diffraction as Ni_3Ti, a stable, coherent, intermetallic compound. The aging temperature influenced the precipitate size and distribution more than the time at temperature. As the aging temperature was increased...they became more widely spaced. The Ni_3Ti particles formed in a Widmanstatten pattern and greatly enhanced the strength of the martensite.

Figure 1. An optical micrograph of the annealed Vasco Max T-250. Mag. 200X

Figure 2. Transmission electron micrograph of the annealed sample showing the lath martensite structure. Mag. 20KX

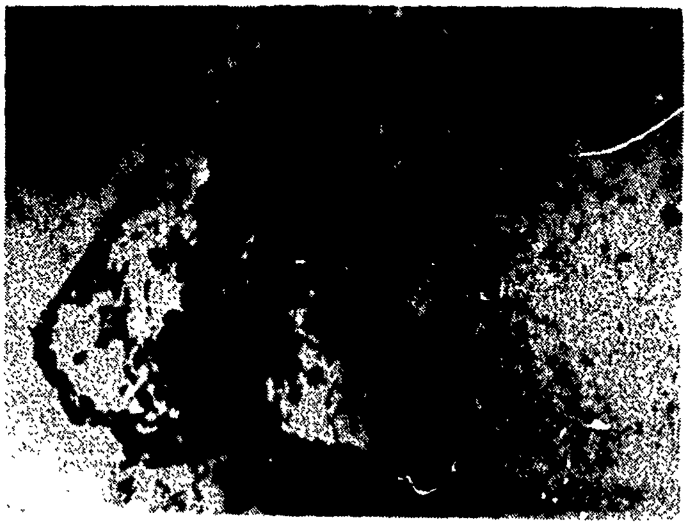


Figure 3. Heavily dislocated lath in the annealed sample. Mag. 50KX

Figure 4. Typical lath martensite structure from the sample aged at 850°F for 3 hours. Mag. 20KX

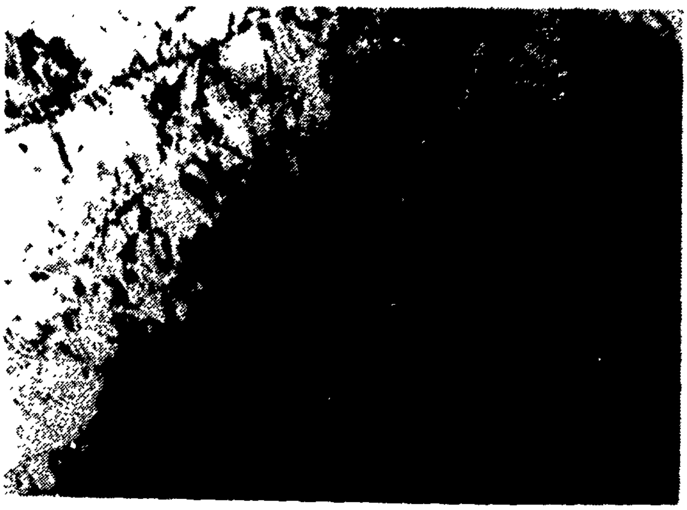
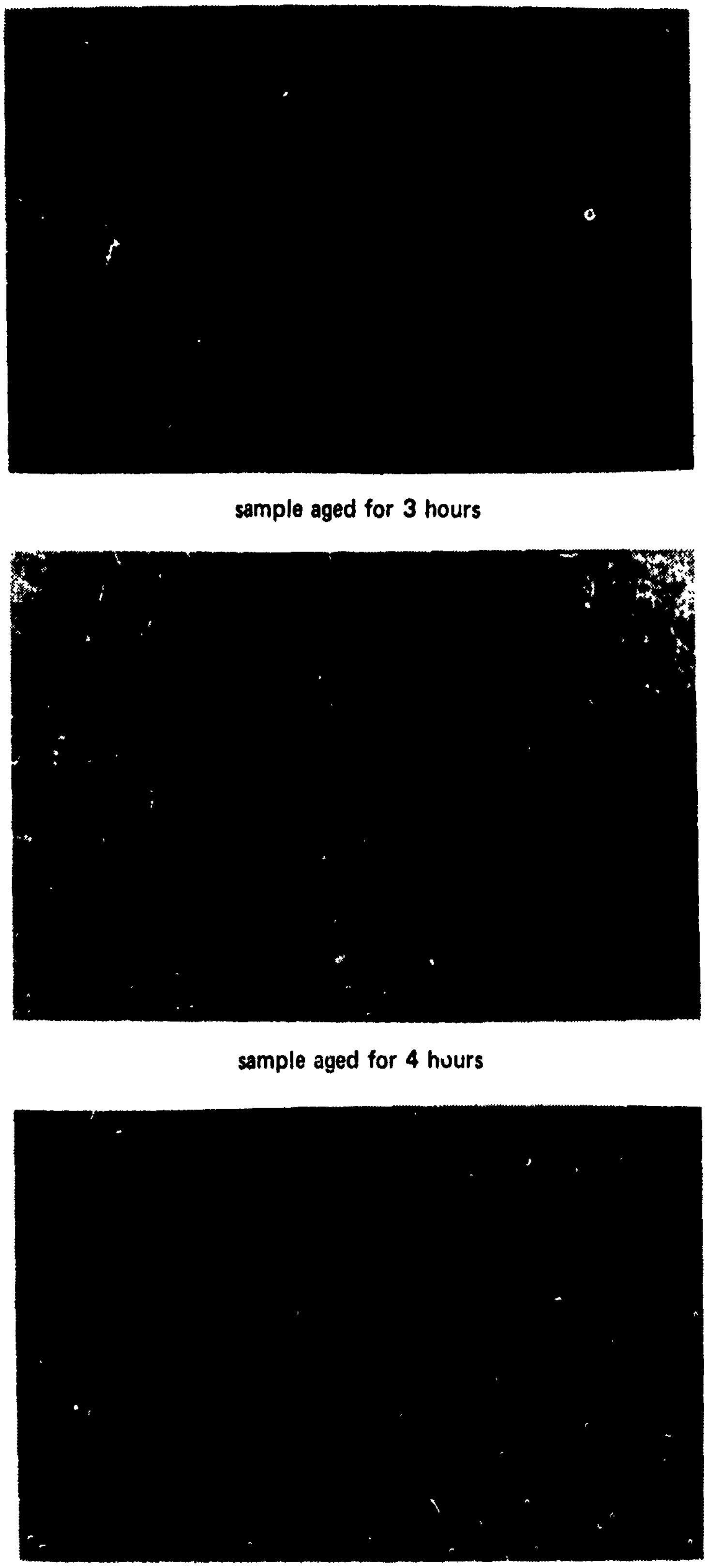


Figure 5. Elongated lath containing a high density dislocation and some second phase particles from the sample aged at 850°F for 3 hours. Mag. 100KX



sample aged for 3 hours

sample aged for 4 hours

sample aged for 8 hours

Figure 6. Dark field micrographs of the samples aged at 850°F for 3, 4, and 8 hours. Mag. 100KX

Figure 7. Lath martensite structure of the sample aged at 900°F for 4 hours. Mag. 20KX

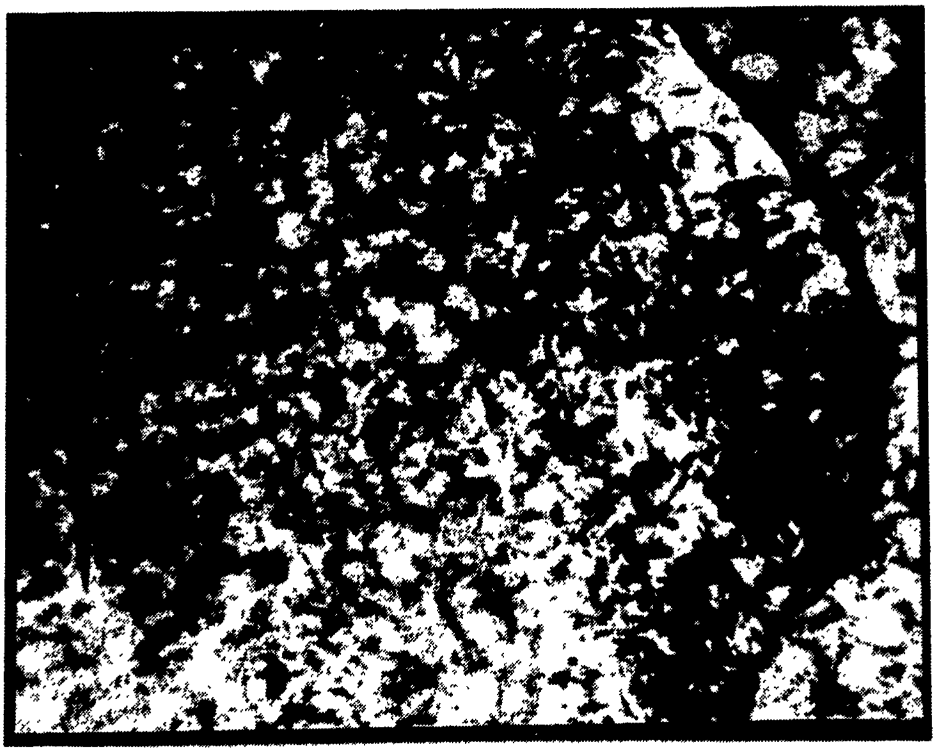


Figure 8. Lath containing second phase particles from the sample aged at 900°F for 4 hours. Mag. 100KX

sample aged for 3 hours

sample aged for 4 hours

sample aged for 8 hours

Figure 9. Dark field micrographs of the samples aged at $900^{\circ}F$ for 3, 4, and 8 hours. Mag. 100KX

Figure 10. Lath martensite structure of the sample aged at 950°F for 4 hours. Mag. 20KX

Figure 11. Electron micrograph of the sample aged at 950°F for 4 hours showing the precipitates. Mag. 100KX

sample aged for 3 hours

sample aged for 4 hours

sample aged for 8 hours

Figure 12. Dark field micrographs of the samples aged at 950°F for 3, 4, and 8 hours. Mag. 100KX

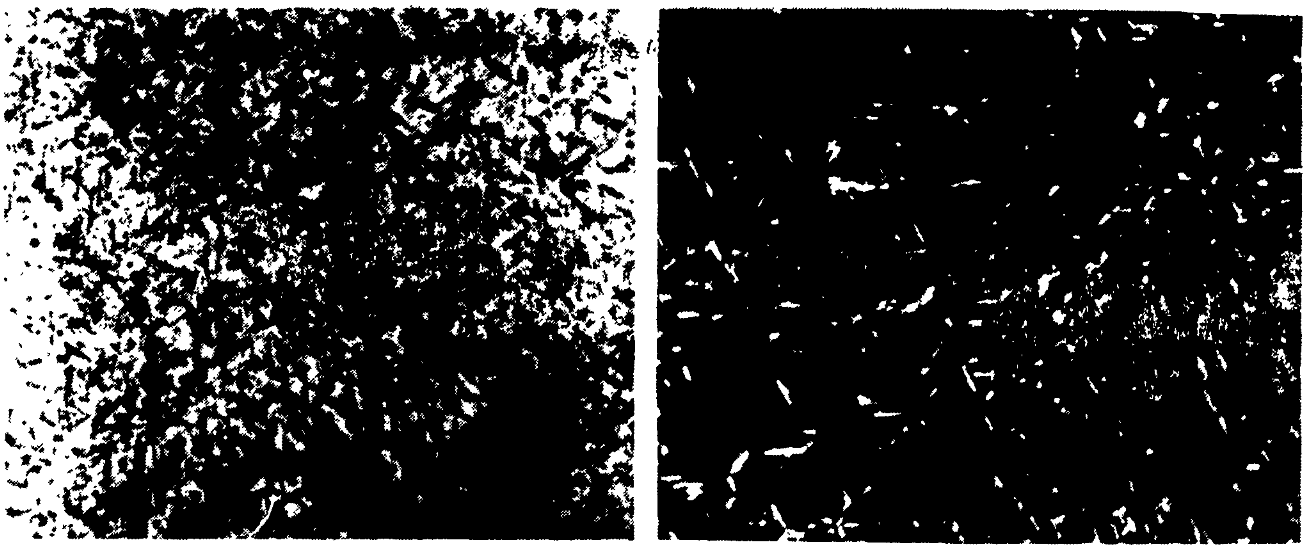


Figure 13. Bright field/dark field pair of the samples aged at 900^{o}F for 3 hours showing the cross sections of the precipitates. Mag. 100KX

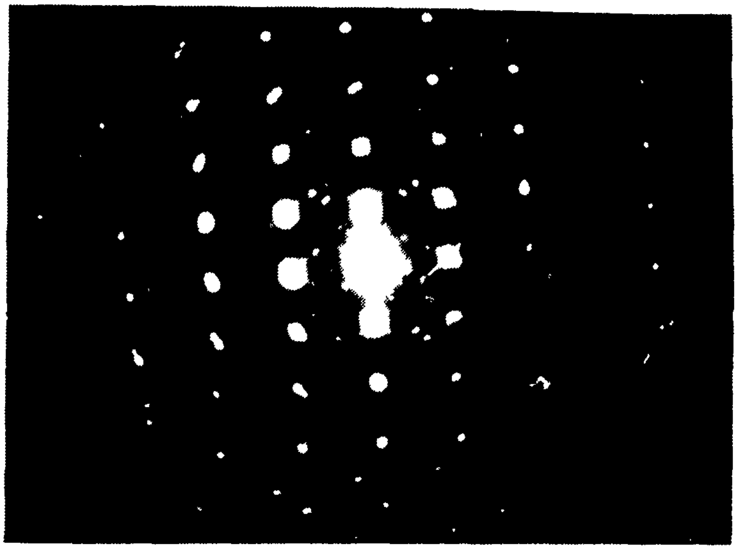


Figure 14. Electron diffraction pattern B = (011) for the matrix and B = (0001) for the precipitates.

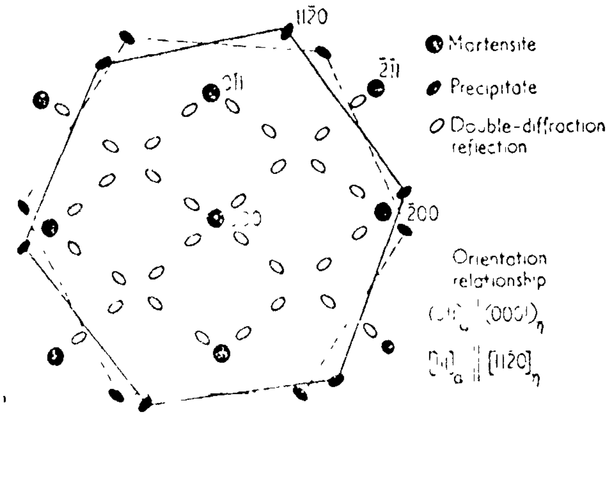


Figure 15. Schematic of the electron diffraction pattern showing the (011) orientation and the (0001) orientation (Ref. 13).

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MICROSTRUCTURAL CHARACTERIZATION OF A
COBALT-FREE MARAGING STEEL, VASCO
MAX T-250 - Kerry N. Siebein

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